IMPROPER FERROELECTRICS

Structural Studies of $LiNH_4SO_4$

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The room temperature crystal structure of LiNH$_4$SO$_4$, complete with the positions of the hydrogen ions, has been determined from neutron diffraction measurements. X-ray diffraction study has shown that the abrupt ferroelectric transition of LiNH$_4$SO$_4$ near 11°C involves a doubling of the orthorhombic unit cell normal to the polar axis. The phonon dispersion in this material above the transition temperature was explored by means of inelastic neutron scattering, but no soft phonon mode associated with the structural transition could be found.

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SUMMARY

The room temperature crystal structure of $LiNH_4SO_4$, complete with the positions of the hydrogen ions, has been determined from neutron diffraction measurements. X-ray diffraction study has shown that the abrupt ferroelectric transition of $LiNH_4SO_4$ near 11°C involves a doubling of the orthorhombic unit cell normal to the polar axis. The phonon dispersion in this material above the transition temperature was explored by means of inelastic neutron scattering, but no soft phonon mode associated with the structural transition could be found.

I.   INTRODUCTION

The class of ferroelectrics known as improper, whose long-range order parameter is a crystal distortion rather than the electric polarization, is currently of considerable scientific and technological interest. According to present understanding, the crystal distortion of such a ferroelectric results from the freezing-in of a non-polar (Brillouin-zone edge) phonon mode, produces a spontaneous ferroelectric polarization.[1,2] The dielectric constant of an improper ferroelectric, unlike that of a proper ferroelectric, does not diverge near the transition temperature, yet its pyroelectric coefficient can remain substantially high. This combination of properties can express itself in a large figure-of-merit for vidicon thermal-imager applications.[3]

The close interplay between the structural and electrical behavior of an improper ferroelectric makes it particularly important that the measured properties of any such material of interest be complemented by detailed investigations of its crystal structure and its static and dynamic structural changes with temperature.

The present studies have focused on the structure of $LiNH_4SO_4$, an improper ferroelectric compound whose thermodynamic and optical properties have recently been explored quite extensively.[3-8] This compound undergoes a ferroelectric order-disorder transition at $T_{c1} \sim 186°C$, above which, in the paraelectric phase, the temperature dependence of its dielectric constant shows

a marked departure from the Curie-Weiss law, indicative of "improper" behavior. At $T_{c2} \approx 11°C$, it transforms abruptly into a different low-temperature ferroelectric state. Thus, between T_{c1} and T_{c2}, the material is in an intermediate ferroelectric phase, which is of particular application interest near room temperature.[3]

The room-temperature crystal structure of $LiNH_4SO_4$ has been previously found to belong to the orthorhombic space group $P2_1cn$.[9] However, since the crystal structure determination was made by x-ray diffraction, the positions of the hydrogen ions could not be located. We have corrected this situation by means of a neutron diffraction study of a $LiNH_4SO_4$ crystal. From our determination of its crystal structure, complete with hydrogen-ion positions, it is now possible to calculate the spontaneous electric polarization of the room-temperature phase of this material on the basis of various model assumptions. Some preliminary calculations are presented, together with our diffraction results for the structure, in Section II of this report.

The structural nature of the transition in $LiNH_4SO_4$, when it is cooled through its lower critical temperature T_{c2}, has recently been explored in an optical Raman study[8], from which it was claimed that the transition involved a doubling of the primitive unit cell. The experimental evidence for this claim, however, cannot be said to be definitive. We have therefore investigated this question with x-ray diffraction measurements, and our results

described in Section III show unambiguously that the unit cell does indeed double along a symmetry axis when the material transforms into its low-temperature phase. Moreover, as part of our neutron diffraction work on this compound and its deuterated variant, we have studied their structural transformation as a detailed function of temperature near T_{c2}. As described in Section III, our neutron diffraction data confirm that the transformation is first-order (occurring at a slightly lower temperature in the deuterated compound) and that it involves the structural changes revealed by our x-ray measurements.

It is quite conceivable that, at temperatures just above the lower critical point T_{c2}, there would occur a softening of a phonon mode, which anticipates dynamically the static structural change (i.e., the unit-cell doubling) that ultimately takes place at T_{c2}. Although the abruptness of the transition would disallow the mode softening from growing continuously all the way to completion, it was nevertheless thought that at least the starting stages of a phonon instability would be detectable in a sensitive measurement. We have therefore carried out a fairly intense survey of inelastic neutron scattering measurements on $LiNH_4SO_4$ and $LiND_4SO_4$ crystals. Our search for soft phonons was ultimately unsuccessful due to an apparent disappearance of phonon scattering near the Brillouin-zone boundary. We nevertheless did obtain some potentially very useful phonon-dispersion information. This work is discussed in Section IV.

II. THE CRYSTAL STRUCTURE OF $LiNH_4SO_4$ AT ROOM TEMPERATURE

The room-temperature neutron diffraction experiments on $LiNH_4SO_4$ crystals were performed on the 4-circle diffractometer at the CP-5 reactor of the Argonne National Laboratory, in collaboration with Dr. Selmer W. Peterson of the Argonne staff. The two single-crystal samples used in these experiments were supplied to us by the Philips North American Corporation and were approximately 10 mg and 70 mg in weight. With a monochromatic incident neutron beam, integrated scattering intensity data were collected for over one hundred Bragg reflections, for which background corrections were made on the basis of individual rocking curves. The data taken on the two crystals for the reflections of weak and moderate intensities were in excellent agreement. For the strong reflections, however, reliable intensity information could only be obtained with the smaller crystal for which extinction effects were very small.

Fourier maps made of all our neutron scattering data verified that the crystal structure of $LiNH_4SO_4$ belongs to the space group $P2_1$ cn with orthorhombic unit cell dimensions, a = 5.280 Å, b = 9.140 Å, c = 8.786 Å, as determined earlier by Dollase from x-ray diffraction measurements.[9] However, our results extended beyond those of the x-ray work in that they were able to locate the positions of the hydrogen atoms. As shown in Fig. 1, the four hydrogens of each NH_4 complex form a slightly distorted tetrahedron with the nitrogen essentially at the geometric center, the N-H distances varying from 0.938 Å to 0.996 Å. The H-N-H

angles vary between about 106° and 112°, the ideal tetrahedral angle being 109.5°. The orientation of each NH_4 tetrahedron is such that the hydrogens are placed in close proximity with the nearest oxygens of the surrounding network of SO_4 and LiO_4 tetrahedra. Thus, the existence of strong H-O bonds is clearly manifested in the structure. Specifically, the H-O bonds are oriented such that two of the hydrogen atoms are approximately on the perpendicular-bisecting plane of the line joining neighboring oxygen pairs. Moreover, the thermal vibration ellipsoid for these hydrogen atoms is highly eccentric, with its long axis lying in this plane. The other two hydrogen atoms have only one oxygen neighbor and their thermal vibration ellipsoids are nearly spherical.

The room-temperature crystal structure of $LiNH_4SO_4$ having thus been determined completely, we proceeded to calculate its spontaneous ferroelectric polarization. For these calculations, we adopted a point-charge model in which the ionic valences were set to be Li^+, S^{6+}, and O^{2-}, while the NH_4 complexes were considered in several alternative cases. In the first case we assumed H^+ and N^{3-}, and in the second case H^- and N^{5+}. The calculated polarizations were respectively 241.6 and 157.0, in $\mu C/cm^2$ directed parallel to the a axis (see Fig. 1). We also considered the case of H^0 and N^+, and obtained the intermediate polarization value of 198.7 $\mu C/cm^2$, again parallel to a. All three of these

calculated values for the room-temperature spontaneous polarization of $LiNH_4SO_4$ are in gross disagreement with the experimentally determined values of ~0.3 $\mu C/cm^2$ [5] and ~0.8 $\mu C/cm^2$ [7]. However, at least one of these two sets of experimental results may well be suspect since they disagree on the sign of the abrupt polarization change at the 11°C transition. Nevertheless, the enormous discrepancy between the calculated and experimental values for the room-temperature polarization is probably real and most likely arises from our simplistic assumption of a completely ionic model in which no allowance was made for any degree of covalency resulting in some charge distributed in the ligands. Such an allowance, however, would require information about the electronic wavefunctions of $LiNH_4SO_4$ that is presently unavailable.

III. THE STRUCTURAL NATURE OF THE LOW-TEMPERATURE FERROELECTRIC
TRANSITION IN $LiNH_4SO_4$

X-ray diffraction experiments were undertaken in order to investigate the structural changes that may accompany the abrupt ferroelectric transition in $LiNH_4SO_4$ at ~11°C. This work also was carried out in collaboration with Dr. S. W. Peterson at the Argonne National Laboratory. For these experiments, a small piece taken from the larger crystal used in our neutron diffraction studies was mounted on a cryogenic stage attached to

an x-ray precession camera. For studies below the transition point, the sample temperature was maintained at -1.5°C by means of a steady flow of the cool vapor of liquid nitrogen. At this temperature and at room temperature, precession photographs were taken along the orthorhombic a*, b*, and c* axes in various reciprocal-lattice planes. The results obtained in the (h0ℓ) planes are shown superimposed for the two temperatures in Fig. 2. It is clear from this figure that a new set of spot images appeared at -1.5°C, which have one-half the spacing along $\vec{c}*$ of those observed at room temperature. Specifically, from the numerical values shown for the principal spacings: at -1.5°C the ratio a*/c* $\approx$ 8.1/2.4 $\approx$ 3.37, whereas at room temperature a*/c* $\approx$ 8.1/4.8 $\approx$ 1.69. Thus, the unit cell in real space doubles along the c axis (i.e., normal to the polar a axis) when the material is cooled through its ferroelectric transition at $\sim$11°C. This is precisely the structural change that was suggested earlier on the basis of optical measurements.[8] Furthermore, our results appear to indicate that the orthorhombic symmetry is preserved during this structural change. However, we find that the intensities and absences of the various reflections for the low temperature phase cannot be explained on the basis of any orthorhombic space group. We believe that this difficulty probably arises from the existence of crystallographic twins of presumably lower symmetry. If this is so, very careful work will be needed to suppress the twinning and allow the low temperature structure to be identified.

While carrying out neutron scattering measurements of the phonon dispersion in a $LiNH_4SO_4$ crystal and a deuterated ($LiND_4SO_4$) crystal, which will be discussed in the following section, we monitored the intensities of selected Bragg reflections as the temperature was varied through the ferroelectric transition point. In the case of the undeuterated crystal, the (003) reflection was chosen, which is forbidden for the room temperature phase. As shown in Fig. 3, the intensity of this reflection increased abruptly at $\sim 9°C$ as the temperature was lowered and then decreased with similar abruptness at $\sim 12°C$ as the temperature was subsequently raised. These data support our x-ray diffraction results for the structural change that accompanies this transition and also show that the change takes place essentially discontinuously with a sizable temperature hysteresis. Thus, the ferroelectric transition in $LiNH_4SO_4$ is unambiguously first-order, which is consistent with previous reports based on dielectric measurements.[5,7] In the case of the deuterated crystal, both the (200) and (20 1/2) reflections (the latter indexed as (201) for the low temperature phase) were monitored in intensity as the temperature was slowly raised through the critical region. It was observed that while the (200) intensity exhibited virtually no change, the (20 1/2) intensity dropped discontinuously at $\sim 7°C$, as shown in Fig. 3. These data are also consistent with our x-ray diffraction results, indicating that $LiND_4SO_4$ and $LiNH_4SO_4$ undergo the same crystallographic

change at their ferroelectric transition. Furthermore, the tran-
sition in $LiND_4SO_4$ is also first-order but occurs at a slightly
lower temperature, which again agrees with the results of previous
dielectric measurements.[4]

IV. SEARCH FOR SOFT PHONONS IN $LiNH_4SO_4$ AND $LiND_4SO_4$

    Inelastic neutron scattering measurements were initially
performed on a $LiNH_4SO_4$ crystal at the CP-5 reactor of the Argonne
National Laboratory. Having found that the structural change in
$LiNH_4SO_4$, when cooled through its ferroelectric transition point,
involves a unit-cell doubling along the orthorhombic c axis, our
search for a soft phonon mode just above the transition point was
concentrated in the reciprocal-space region of the strongest
superlattice reflections, such as (20 1/2). This region core-
sponds to that of the Brillouin-zone boundary where the wavevector
$\vec{k} = 1/2 \; \vec{c}*$. Thus, energy scans were made, typically up to ∿8 meV,
with the neutron scattering vector $\vec{q}$ parallel to $\vec{c}*$ and fixed in
magnitude at some value between zero and 1/2 c* ($=\pi/c$), as meas-
ured from the reciprocal lattice point. However, due to an
enormous q-dependent background produced by incoherent scattering,
inelastic scattering peaks attributable to phonons could only be
detected with difficulty.

Since the large incoherent scattering was thought to arise primarily from the hydrogen, the $LiNH_4SO_4$ crystal sample was replaced by a deuterated ($LiND_4SO_4$) crystal. The sample replacement was extremely effective in reducing the scattering background, and it immediately became much easier to observe the room-temperature inelastic scattering from a transverse acoustical phonon branch (with polarization parallel to $\vec{a}$) but only at q values up to ∿0.2c*. At larger values of q, where this phonon branch extends to higher energies (E), the intensity of the inelastic scattering peak was seen to diminish as $1/E^2$ (as expected theoretically) and thus to rapidly become unmeasurable.

In order to follow this acoustical phonon branch closer to the Brillouin zone boundary and to observe any other phonon modes at higher energies, we needed access to a more intense neutron source. Hence, the $LiND_4SO_4$ crystal was transferred to the high-flux reactor at the Brookhaven National Laboratory, where the remainder of our inelastic scattering experiments were performed. With an order-of-magnitude increase of incident neutron flux, the transverse acoustical (TA) phonon branch seen at Argonne was followed out to q ∿ 0.3c*, again at room temperature. Beyond this value of q, there was an anomalously rapid decrease in the observed intensity of the inelastic scattering peak, making it impossible to learn if this phonon branch ultimately softens (i.e., dips to lower energies) near the Brillouin-zone boundary. Up to q ∿ 0.3c*, the phonon dispersion for this TA branch is

linear, as shown in Fig. 4, corresponding to a sound velocity (v_s) of $\sim 2.2 \times 10^5$ cm/sec. Moreover, as indicated in the figure, there was no apparent change in this phonon dispersion curve when the sample temperature was lowered first to 7.5°C and then to - 40°C. At the latter temperature, we also observed a longitudinal acoustical phonon branch at higher energies (see Fig. 4), but again the inelastic scattering peak was found to disappear when q reached about half-way toward the zone boundary; in this case, the linear dispersion at low q gives $v_s \approx 4.9 \times 10^5$ cm/sec. There seems to be no reasonable explanation for the disappearance of the scattering from both these phonon branches near the zone boundary; further work is needed to clarify this perplexing result.

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FIGURE CAPTIONS

Fig. 1.   The crystal structure of $LiNH_4SO_4$ at room temperature,
          shown projected on the a-b plane, where the dotted
          lines define half the orthorhombic unit cell. The
          elevations of the atoms are indicated in fractional
          units of c, except for the Li atoms, which lie approxi-
          mately 1/2 c above the S atoms. Only one of the two
          NH_4 complexes in this half-cell are represented (see
          Ref. 9).

Fig. 2.   Composite representation of x-ray precession photographs
          obtained for the (h0ℓ) planes of $LiNH_4SO_4$ at -1.5°C and
          20°C. The ℓ values shown are appropriate for the low-
          temperature phase and are twice those of the room-
          temperature phase.

Fig. 3.   Neutron Bragg intensities (in arbitrary units) of the
          (003) reflection from $LiNH_4SO_4$ and of the (20 1/2)
          reflection from $LiND_4SO_4$ for increasing or decreasing
          temperature.

Fig. 4.   Inelastic neutron scattering results for the phonon dis-
          persion (energy in meV vs wavevector parallel to $\vec{c}*$)
          in $LiND_4SO_4$, for a transverse acoustical (TA) mode with
          phonon polarization parallel to $\vec{a}*$ and for a longitudinal
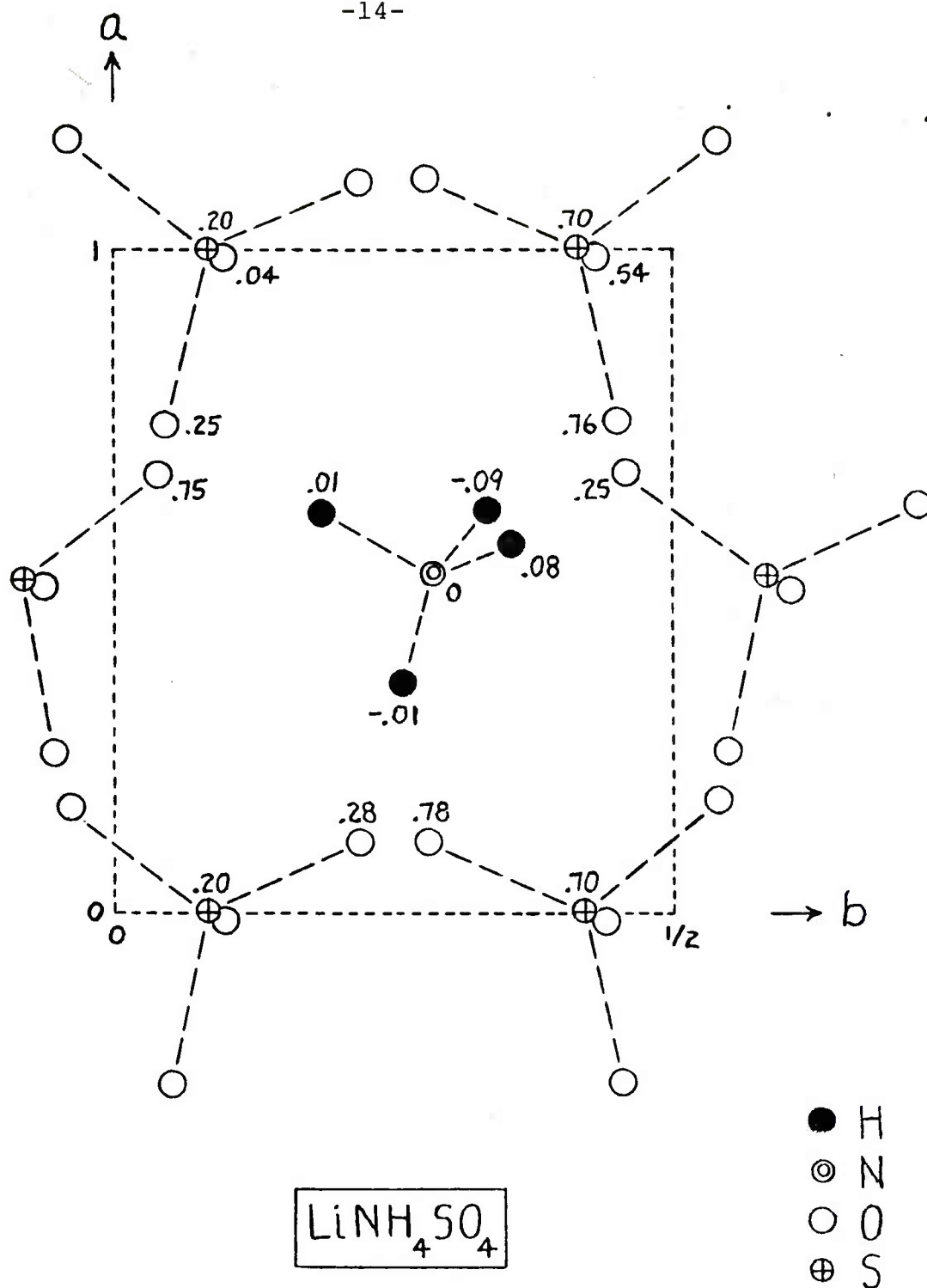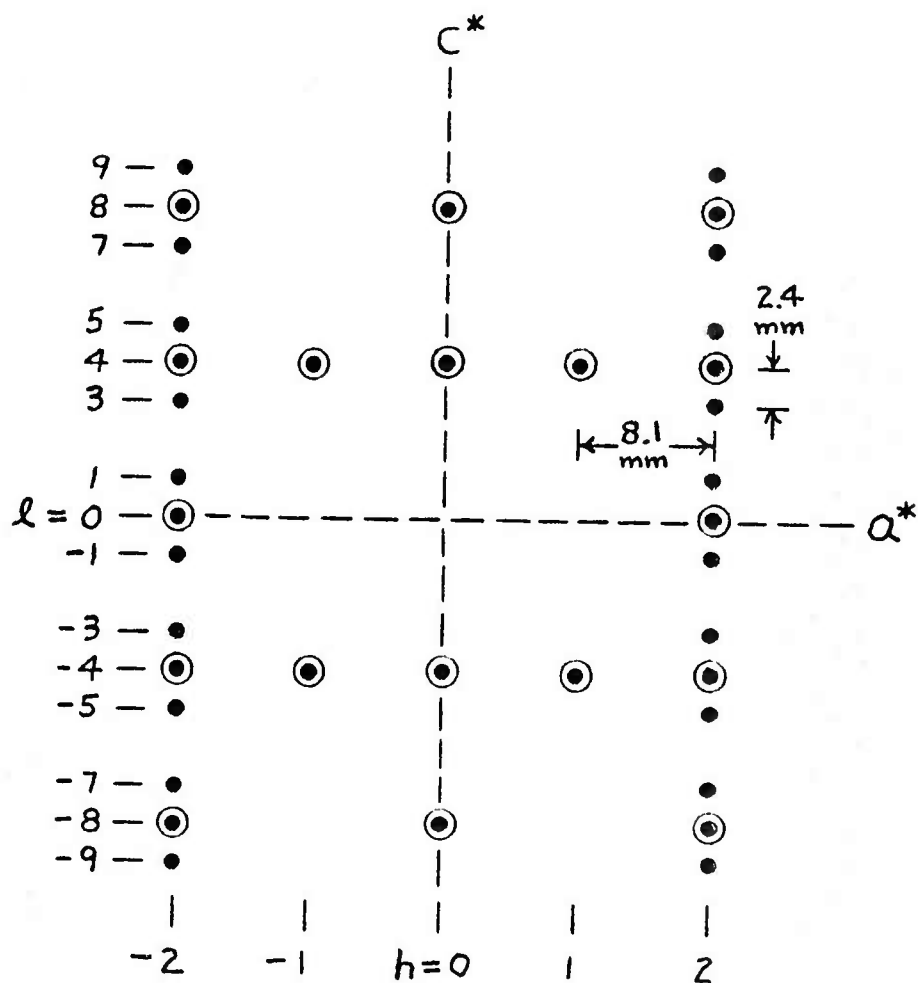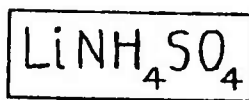   accoustical (LA) mode, at various temperatures.

Figure 1

$$\boxed{LiNH_4SO_4}$$

• image observed only at -1.5°C

◉ image observed at -1.5°C and 20°C
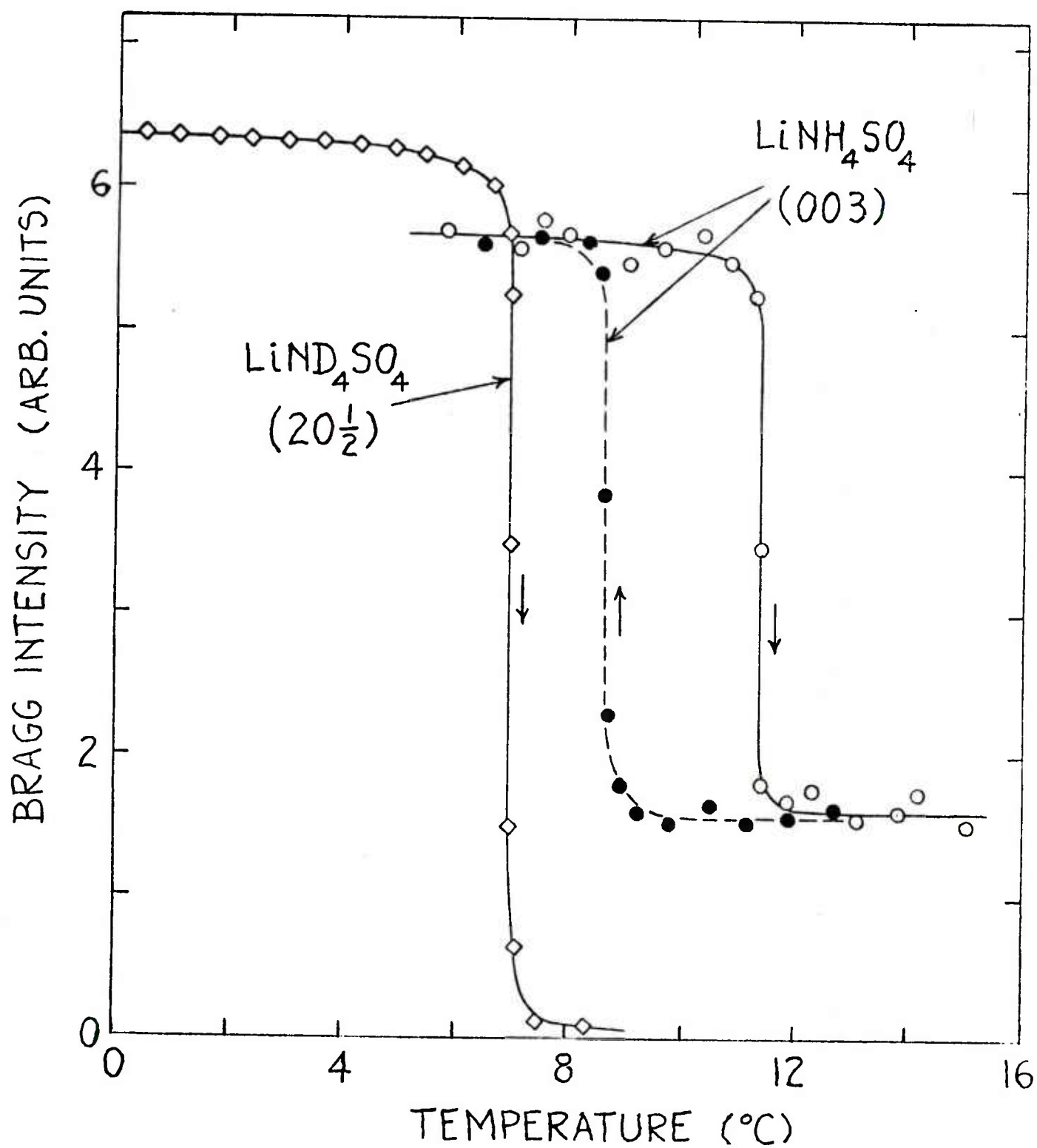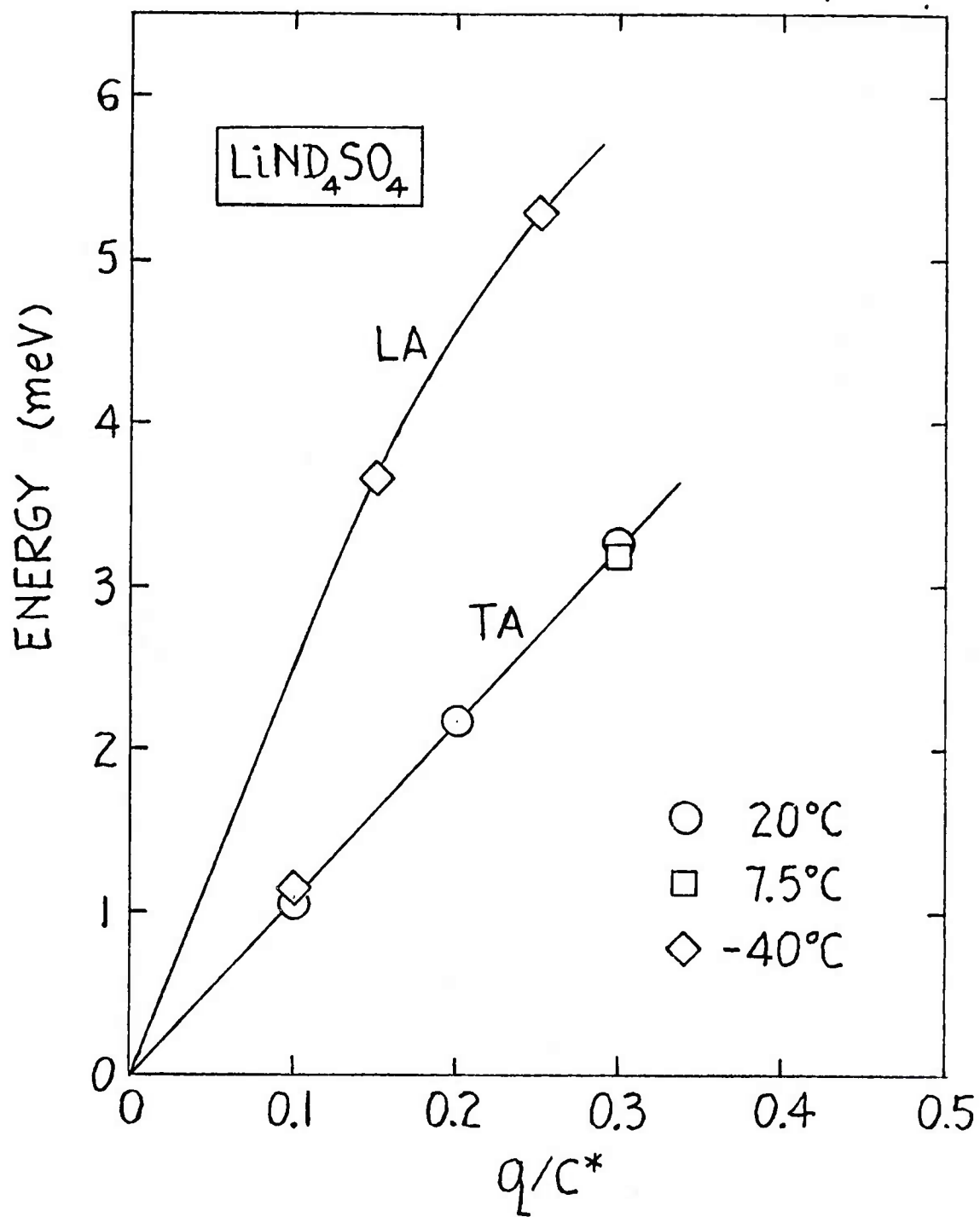
Figure 2

Figure 3

Figure 4

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